IDEM **Guidance**

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SOLID WASTE PROGRAM

ANALYTICAL DATA

DELIVERABLE

REQUIREMENTS:

A GUIDANCE DOCUMENT

Indiana Department of Environmental Management
Office of Land Quality
Chemistry Section

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ANALYTICAL DATA DELIVERABLE REQUIREMENTS: A GUIDANCE DOCUMENT

Purpose

The purpose of this document is to provide guidance to facilities submitting analytical data in support of the Solid Waste Program, as defined by 329 IAC 10, 11, and 12.

The Office of Land Quality (OLQ) of the Indiana Department of Environmental Management (IDEM) has established the minimum requirements for Analytical Data Quality (ADQ) that clearly define the reporting and deliverables required to verify data validity.

ADQ Levels

The ADQ Levels, as recognized by OLQ, are distinguished by the types of technology and documentation used, and their degree of sophistication as follows:

Level I - Field screening. This Level is characterized by using portable instruments that can provide real-time data to assist in the optimization of sampling point locations and for health and safety support. Level I is only used for qualitative analyses; it is not to be used for quantitative analyses. Most portable instruments require calibration.

Level II - Field analysis. This Level is characterized by using portable analytical instruments that can be used on-site. This Level is considered semi-quantitative due to the lack of supporting Quality Assurance/Quality Control (QA/QC) documentation. All portable analytical instruments require calibration.

Level III - This Level is designed to provide laboratory analysis using EPA and other recognized standard procedures with rigorous QA/QC protocols. Documentation provides confirmed identification and quantification of compounds in environmental samples.

Level IV - This Level is characterized by rigorous QA/QC protocols and documentation. It also provides qualitative and quantitative analytical data of defensible quality.

Level V - These are non-standard methods that will require pre-approval for use. These are analyses that may require modification and/or development. Level V requirements include Level IV requirements plus any additional documentation needed to assess method modifications.

ADQ Levels for the analytical data required by the Solid Waste Program will vary depending on the type of project and potential risk to human health and the environment. The following lists provide the minimum ADQ deliverable levels for the projects specified.

PROJECTS REQUIRING LEVEL III ADQ DELIVERABLES

Associated with permitted disposal facility (except PCBs)
- Ground water monitoring data
- Special waste certification
- Restricted waste classification

Encapsulated waste for legitimate use
Encased waste (unless potential ecological impact, predicated by engineering design)
Alternative daily cover data
Data for treatment verification

PROJECTS REQUIRING LEVEL IV ADQ DELIVERABLES

Direct human contact
Direct contact with surface waters
Potential ecological risk
PCB data
Sediment data
Enforcement / legal case data
Demonstration projects
Risk assessments
All other legitimate uses

Note: **The OLQ reserves the option to require that all raw data and any other relevant information be submitted if data validity concerns arise during the review of the analytical data packages.**

There are no specific projects where Level I, II, or V deliverables are currently required by the Solid Waste Program.

Methods and Procedures

Analytical considerations must be evaluated concurrently with statistical and sampling considerations to ensure that established ADQ can be attained. Facilities must determine the sampling methods, analytical methods, and quality control measures needed to meet the ADQ requirements established in this document. Commonly, the analytical method indicates the quality control required to validate the results of that method. Analyses must comply with

applicable QA/QC procedures. Guidance for QA/QC measures and documentation can be found in:

- "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, Third Edition (September 1986), Final Update 1 (July 1992), Final Update 2 (September 1994) and 2A (April 1995), and Final Update 2B (April 1995);
- Chapter One of SW-846, Third Edition, Final Update 1, July 1992;
- "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, March 1983;
- "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA 600/R-93/100, August 1993; and
- Other methods approved by the commissioner.

Note that in some cases a specific sampling or analytical method is required by rule. For example, SW-846 methods are mandatory for :

- Hazardous Characterization,
- Free Liquids Determination,
- Waste Delisting, and
- Incinerator Trial Burns.

Data Validation

The IDEM must confirm that analytical data is valid by reviewing the QA/QC data generated during the sampling and analysis procedures. This will assure that scientifically sound decisions are made which will be protective of human health and the environment. The QA/QC data necessary for validation is required to be submitted with analytical data packages.

Sampling and analytical documentation is a required element of data packages in order to validate data quality. Reporting and deliverable requirements for Level III ADQ data packages are listed on the following pages. **Level IV ADQ requires the same reporting and deliverables as the Level III ADQ, but must also include all raw data. Chromatograms, recorder outputs, mass spectrum reports, computer printouts, charts, graphs, bench sheets, or any other hard copy data generated during sampling and analysis are components of the raw data.**

The OLQ reserves the option to require that all raw data and any other relevant information be submitted if data validity concerns arise during the review of the analytical data packages.

SAMPLING DOCUMENTATION

Specific procedures describing how the sampling operations were actually performed should be provided. A simple reference to standard methods is not sufficient unless a procedure was performed exactly as described in the published method. Methods from source documents published by the EPA, American Society for Testing and Materials (ASTM), U.S. Department of the Interior, National Ground Water Association, American Petroleum Institute (API), or other

recognized organizations with appropriate expertise should be used, if possible. The procedures for sample collection should be documented by including at least the following:

- Applicability of the procedure,

- Equipment used,

- Measures used to ensure that representative samples were collected, and

- Detailed description of procedures that were followed in collecting the samples.

ANALYTICAL DOCUMENTATION - Level III ADQ

The analytical documentation should include an organized summary of the final results, a copy of signed chain-of-custody for each sample, and all quality control documentation, including a report case narrative that explains any QA/QC or analysis problems encountered and the corrective actions taken.

Specifically, analytical documentation should include the following for each type of analysis:

I. Metals by Atomic Absorption Spectroscopy

A. Documentation of analysis dates and methods showing:

1. Sampling date,
2. Facility sample number and lab sample number,
3. Preservative used (when applicable),
4. Extraction date (when applicable),
5. Digestion date,
6. Analysis date and time of day,
7. Extraction, digestion, and analytical method numbers,
8. Report date, and
9. Chain-of-custody report.

B. Results of method and lab blanks, including detection limits.

C. Results of lab replicates (when applicable).

D. Results of instrument calibration (three-point or five-point) documented by:

1. Calibration curve for each metal,
2. Correlation coefficient for each metal, and
3. Standard deviation and relative standard deviation.

E. Initial calibration verification and continuing calibration verification documented by :

1. Results of verification standard analysis (concentration of standard) and

2. Percent recoveries.

F. Results of matrix spike/matrix spike duplicate analysis documented by:

1. Concentration of analyte in original sample (before spiking),
2. Amount of spike for each component,
3. Spiked sample result for each component,
4. Percent recovery of each component,
5. Relative percent difference between spike and spike duplicate of each component, and
6. Analysis date and time.

G. Results of Method of Standard Additions (when applicable).

H. Results of Laboratory Control Sample.

I. Toxicity Characteristic Leaching Procedure (TCLP) and Leaching Method (Neutral) results (when applicable) including:

1. Sample preparation records,
2. Extraction records,
3. Determinative analysis records, and
4. Calculations.

J. Detection limit summary report.

K. Holding times summary report.

II. Metals by Inductively Coupled Plasma Spectroscopy

A. Documentation of analysis dates and methods showing:

1. Sampling date,
2. Facility sample number and lab sample number,
3. Preservative used (when applicable),
4. Extraction date (when applicable),
5. Digestion date,
6. Analysis date and time of day,
7. Extraction, digestion, and analytical method numbers,
8. Report date, and
9. Chain-of-custody report.

B. Results of method and lab blanks, including detection limits.

C. Results of lab replicates (when applicable).

D. Results of instrument calibration (three-point or five-point) documented by:

1. Calibration curve for each metal,
2. Correlation coefficient for each metal, and
3. Standard deviation and relative standard deviation.

E. Inductively Coupled Plasma (ICP) linear range report.

F. Initial calibration verification and continuing calibration verification documented by:

1. Results of verification standard analysis (concentration of standard) and
2. Percent recoveries.

G. Interference check sample results.

H. ICP serial dilution results (when applicable).

I. ICP interelement correction factors.

J. Results of matrix spike/matrix spike duplicate analysis documented by:

1. Concentration of analyte in original sample (before spiking),
2. Amount of spike for each component,
3. Spiked sample result for each component,
4. Percent recovery of each component,
5. Relative percent difference between spike and spike duplicate of each component, and
6. Analysis date and time.

K. Results of Method of Standard Additions (when applicable).

L. Results of Laboratory Control Sample.

M. Toxicity Characteristic Leaching Procedure (TCLP) and Leaching Method (Neutral) results (when applicable) including:

1. Sample preparation records,
2. Extraction records,
3. Determinative analysis records, and
4. Calculations.

N. Detection limit summary report.

O. Holding times summary report.

III. General Inorganic Analysis:

A. Provide dates and methods showing:

1. Sampling date,
2. Facility sample number and lab sample number,
3. Preservative used (when applicable),
4. Extraction date (when applicable),
5. Digestion date (when applicable),
6. Analysis date and time of day,
7. Extraction, digestion, and analytical method numbers,
8. Report date, and
9. Chain-of-custody report.

B. Results of method and lab blanks, including detection limits.

C. Results of lab replicates.

D. Results of instrument or standard calibration including:

1. Calibration curve,
2. Correlation coefficient, and
3. Standard deviation and relative standard deviation data (when applicable).

E. Continuing calibration verification including:

1. Results of verification standard (concentration of standard) and
2. Percent recovery.

F. Results of matrix spike/matrix spike duplicate including:

1. Concentration of analyte in original sample (before spiking),
2. Amount of spike for each component,
3. Spiked sample result for each component,
4. Percent recovery of each component,
5. Relative percent difference between spike and spike duplicate of each component, and
6. Analysis date and time.

G. Results of Lab Control Sample.

H. Holding times summary report.

I. Detection limit summary report.

J. Leaching Method (Neutral) results (when applicable) including:

1. Sample preparation records,
2. Extraction records (where applicable),
3. Determinative analysis records, and

4. Calculations.

IV. Volatile and Semi-volatile Organics by Gas Chromatography (GC)

A. Documentation of analysis dates and methods showing:

1. Sampling date,
2. Facility sample number and lab sample number,
3. Extraction date (when applicable),
4. Analysis date and time of day,
5. Extraction and analytical method numbers,
6. Report date, and
7. Chain-of-custody report.

B. Results of five-point external standard calibration documented by:

1. Retention time for each compound,
2. Calibration curve and response factor,
3. Average response factor, and
4. Percent relative standard deviation for each compound in standard.

C. Continuing calibration results documented by:

1. Response factors of each compound,
2. Average response factor from calibration curve for each compound, and
3. Percent difference of each response factor.

D. Summary of surrogate recoveries for each sample.

E. Matrix spike/matrix spike duplicate results documented by:

1. Concentration of analyte in original sample (before spiking),
2. Amount of spike for each component,
3. Spiked sample result for each component,
4. Percent recovery of each component,
5. Relative percent difference between spike and spike duplicate of each component, and
6. Analysis date and time.

F. Results of dual column confirmation (when applicable).

G. Toxicity Characteristic Leaching Procedure (TCLP) results (when applicable) including:

1. Sample preparation records,
2. Extraction records,
3. Determinative analysis records, and
4. Calculations.

H. Blank analysis summary report

I. Holding time summary report.

J. Detection Limit Summary Report.

V. Volatile and Semi-volatile Organics by Gas Chromatography/Mass Spectroscopy (GC/MS):

A. Documentation of analysis dates and methods showing:

1. Sampling date,
2. Facility sample number and lab sample number,
3. Preservative used (when applicable),
4. Extraction date (when applicable),
5. Analysis date and time of day,
6. Extraction and analytical method numbers,
7. Report date, and
8. Chain-of-custody report.

B. Method blank summary sheet and results, including detection limits.

C. Results of Bromofluorobenzene (BFB) or Decafluorotriphenylphosphine (DFTPP) tuning criteria.

D. Initial calibration results documented by the following:

1. Total ion chromatogram,
2. Summary of retention times for all target compounds,
3. Response factors for each target compound in the calibration standards,
4. Average response factor for each compound,
5. Percent relative standard deviations for the five concentrations,
6. System performance and calibration check compounds clearly marked, and
7. Date and time of injection.

E. Continuing calibration results documented by:

1. Response factors of each compound in the standard,
2. Average response factor from initial calibration for each compound,
3. Percent difference of each response factor,
4. System performance and calibration check compounds clearly marked, and

5. Date and time of injection.

F. Summary of internal standards for each sample documented by:

1. Area of primary peak for each standard from the 12 hour standard and the respective retention time (RT),
2. Area of primary peak for each standard from each sample and the respective RT, and
3. Upper and lower quality control limits for peak area and RT clearly identified.

G. Summary of surrogate recoveries for each sample.

H. Results of matrix spike/matrix spike duplicate documented by:

1. Concentration of analyte in original sample (before spiking),
2. Amount of spike for each component,
3. Spiked sample result for each component,
4. Percent recovery of each component,
5. Relative percent difference between spike and spike duplicate of each component, and
6. Analysis date and time.

I. Results of Tentatively Identified Compounds (TICs) documented by:

1. Name of TIC (list as unknown if unidentifiable),
2. Estimated concentration using closest internal standard,
3. For volatiles, list first ten (10) TICs (even if unknown), and
4. For semi-volatiles, list first twenty (20) TICs (even if unknown).

J. Toxicity Characteristic Leaching Procedure (TCLP) results (when applicable) including:

1. Sample preparation records,
2. Extraction records,
3. Determinative analysis records, and
4. Calculations.

K. Holding time summary report, including holding time for extracts prior to analysis (when applicable).

L. Detection limit summary report.

VI. Gas Chromatography for PCB/Pesticide/Herbicide Analysis:

A. Documentation of analysis dates and methods showing:

1. Sampling date,

2. Facility sample number and lab sample number,
3. Extraction date,
4. Analysis date and time of day,
5. Extraction and analytical method number(s),
6. Report date, and
7. Chain-of-custody report.

B. Results of external standard initial calibration for:

1. Single component analytes (three-point or five-point calibration) documented by:

a. Calibration chromatograms,
b. Summary of RTs and RT windows for each standard,
c. Summary of calibration factors calculated for each concentration including mean and percent relative standard deviation for each standard, and
d. Percent breakdown of Endrin and DDT.

2. Multi-component analytes (one point calibration allowable if 3 to 5 point has been established) documented by:

a. RTs and RT windows for each major peak of each analyte (at each concentration, if applicable),
b. Calibration factor for each major peak of each analyte (at each concentration,
c. Calibration chromatograms.

C. If internal standards are used, include as documentation:

1. Identification of internal standards used and compounds of interest associated with each, and
2. Summary of RT windows and calibration factors.

D. Continuing calibration results documented by pesticide calibration verification summary including:

1. RT windows and relative percent difference for analytes listed,
2. Percent breakdown of Endrin, DDT, and combined breakdown, and
3. Summary of calibration factors and percent relative standard deviation.

E. Summary of surrogate recoveries for each sample.

F. Matrix spike/matrix spike duplicate results documented by:

1. Concentration of analyte in original sample (before spiking),
2. Amount of spike for each component,
3. Spiked sample result for each component,
4. Percent recovery of each component,
5. Relative percent difference between spike and spike duplicate of each component, and

6. Analysis date and time.

G. Results of dual column confirmation.

H. Toxicity Characteristic Leaching Procedure (TCLP) results (when applicable) including:

1. Sample preparation records,
2. Extraction records,
3. Determinative analysis records, and
4. Calculations.

I. Blank analysis summary.

J. Holding time summary report, including extract holding times (where applicable).

K. Detection limit summary report.

REFERENCES

"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", (SW-846) Third Edition (September 1986), Final Update 1 (July 1992), Final Update 2 (September 1994) and 2A (April 1995), and Final Update 2B (April 1995).

"Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, March 1983.

"Methods for the Determination of Inorganic Substances in Environmental Samples", EPA 600/R-93/100, August 1993.

"Standard Methods for the Examination of Water and Wastewater", 19th Edition, 1995.

"Superfund Data Quality Objectives: Fact Sheet, Interim Final Guidance and Workbook", EPA A540-R-93-071, September 1993.